

## PROPOSED BOD REQUIREMENTS

Activity, QC Element and Details		Cross Reference	Page
<b>Proficiency Testing</b>			
	Renewal: 1 annually, analyzed between 9/1 and 8/15	NR 149.24(3)(a & b)	31-32
	If must report CBOD, laboratory may analyze same PT for CBOD	NR 149.25(3)	32
<b>Initial Demonstration of Capability</b>			
	Exempted if combination of 4 GGAs, PTs (reference samples) and QCS (blinds) tested in year prior to effective date of revision	NR 149.36(3)(a)1	36
<b>Quality Manual</b>			
	General provisions, format, content and revisions	NR 149.37	37-38
<b>Standard Operating Procedures and Analytical Methods Manual</b>			
	SOPs can be copies of published methods followed exactly, with changes and additions specified or developed in-house	NR 149.40(1)	41
	Analytical methods manual can be list of methods followed exactly or modified methods or in-house SOPs; can be part of QA manual	NR 149.40(2)	41-42
<b>DO Meter Calibration</b>			
	Air-Saturated Water, Water-Saturated Air, Winkler or Iodometric Titration	NR 149.44(6)(d)1	45
<b>pH Meter Calibration</b>			
	2 buffer solutions	NR 149.44(6)(d)2	45
<b>Initial Calibration Verification</b>			
	Exempted	NR 149.44(6)(i)1	46
<b>Continuing Calibration Verification</b>			
	Cannot spike DO, not required		
<b>Limits of Detection and Quantitation</b>			
	Exempted	NR 149.48(2)(a)1	54
<b>Method Blank</b>			
	Dilution Water, 1/preparation batch	NR 149.48(3)(a & b)	55
	Detection, control limits and failures	NR 149.48(3)(c & d)	55
<b>Laboratory Control Sample</b>			
	1/preparation batch, GGA as specified in method	NR 149.48(4)(a & b)	55
	Calculations, control limits and failures	NR 149.48(4)(g-i)	56
<b>Matrix Spike and Matrix Spike Duplicate</b>			
	Exempted	NR 149.48(5)(a)3 note	56
<b>Replicates</b>			
	Only required if specified in method	NR 149.48(6)(b)	57
	1/preparation batch and quality system matrix	NR 149.48(6)(c)	57
	Calculations, control limits and failures	NR 149.48(6)(d & e)	57
<b>Quality Control Samples</b>			
	Language needs clarification to address special case of BOD	NR 149.48(8)	58
<b>Documentation and Labeling of Standards &amp; Reagents</b>			
	Example: GGA source, concentration, purity, manufacturer, lot number, date of receipt and expiration	NR 149.45(1-2)	49
<b>Support Equipment</b>			
	Thermometer, NIST-traceable, annual verification	NR 149.44(3)(d)	43
	Record incubator temperature on testing days and set to maintain proper temperature non-working (weekends/holidays) hours	NR 149.44(3)(e & f)	43-44
	Analytical balance, verified monthly in gm and mg-range	NR 149.44(3)(g)	44
	Weights, NIST-traceable, 3-year external certification	NR 149.44(3)(g)1 & 2	44
<b>Sample Collection, Handling, and Storage</b>			
	Sample collection records	NR 149.46(1)(a)	49
	Container cleanliness	NR 149.46(1)(b)	50
	Sample acceptance policy	NR 149.46(2)	50
	Sample handling protocols	NR 149.46(3)	50-52
	Sample storage	NR 149.46(4)	52
<b>Data Reporting</b>			
	Reported directly to WDNR	NR 149.47(1)(d)1	52
	Report elements, if not reported directly to WDNR	NR 149.47(1)(e)	52-53

### SAMPLE BOD BENCHSHEET

Sample Location	Type	Date	Sample Time					Date in: 12/2/04		Date out: 12/7/04					
Influent	24-hr Comp	12/2/04	6:55 AM					Time in: 8:05 AM		Time out: 8:10 AM					
Effluent	24-hr Comp	12/2/04	6:30 AM					Incubator Temp: 21		Incubator Temp: 20					
Sample ID	Bottle ID	Sample mLs	CBOD	Seed, mLs	pH	Cl <sub>2</sub>	Temp	DO, initial	DO, final	Difference	Seed Correction	BOD (mg/L)	Average BOD	Reported BOD, mg/L	Comment
Blank	1						19.8	8.9	8.8	0.1		0.1			OK
Seed Control	10	5						8.9	6.7	2.2		0.44			
Seed Control	11	10						8.9	5.5	3.4		0.34			
Seed Control	12	12						8.8	4.3	4.5		0.38			
LCS (GGA)	23	6	2					8.9	3.3	5.6	0.8	239.5		240	Qualify LCS
Influent 12-2-04	A	3		2	7.2	No	19.8	8.8	6.8	2.0		200.0	203.0	203	
Influent 12-2-04	BC	5		2	7.2	No	19.8	8.8	5.2	3.6		216.0			
Influent 12-2-04	D	7		2	7.2	No	19.8	8.9	4.4	4.5		192.9			
Effluent 12-2-04	E	200		2	7.3	No	21.5	8.8	6.8	2.0		3.0	2.6	3	
Effluent 12-2-04	GG	250		2	7.3	No	21.5	8.8	6.6	2.2		2.6			
Effluent 12-2-04	H	300		2	7.3	No	21.5	8.8	6.5	2.3		2.3			

**DO Meter Calibration:**

Test Start Meter Calibration Technique	Date: 12/2/04	Time: 7:15 AM	Analyst: Joe Q. Public
<input checked="" type="checkbox"/> Water-Saturated Air DW Temp (°C): 19.8 Pressure (mm Hg): 740 DO Calibration: 8.86	<input type="checkbox"/> Air-Saturated Water DW Temp (°C): Pressure (mm Hg): DO Calibration:		<input type="checkbox"/> Winkler Titration mL Titrant: DO Calibration:
Test End Meter Calibration Technique	Date: 12/7/04	Time: 8:00 AM	Analyst: Joe Q. Public
<input checked="" type="checkbox"/> Water-Saturated Air DW Temp (°C): 20.2 Pressure (mm Hg): 720 DO Calibration: 8.81	<input type="checkbox"/> Air-Saturated Water DW Temp (°C): Pressure (mm Hg): DO Calibration:		<input type="checkbox"/> Winkler Titration mL Titrant: DO Calibration:

Comments: GGA result, 240 mg/L, exceeds upper limit of 228.5 mg/L. Laboratory will use new GGA for LCS next week.

## PROPOSED AMMONIA (ISE) REQUIREMENTS

Activity, QC Element and Details	Cross Reference	Page
<b>Proficiency Testing</b>		
Renewal: 1 annually, analyzed between 9/1 and 8/15	NR 149.24(3)(a & b)	31-32
<b>Initial Demonstration of Capability</b>		
Exempted if combination of 4 LCS, MS, MSDs, PTs, QCS and Replicates analyzed in year prior to effective date	NR 149.36(3)(a)1	36
<b>Quality Manual</b>		
General provisions, format, content and revisions	NR 149.37	37-38
<b>Standard Operating Procedures and Analytical Methods Manual</b>		
SOPs can be copies of published methods followed exactly, with changes and additions specified or developed in-house	NR 149.40(1)	41
Analytical methods manual can be list of SOPs; part of QA manual	NR 149.40(2)	41-42
<b>Ion Selective Electrode Meter Calibration, Source 1</b>		
2 standards, minimum, daily	NR 149.44(6)(d)2	45
Nernst equation; $R \geq 0.995$ for linear	NR 149.44(6)(f)1 & (g)2	45-46
<b>Initial Calibration Verification, Source 2</b>		
Exempted	NR 149.44(6)(i)1	46
<b>Continuing Calibration Verification, Source 1</b>		
One, at end of batch and after 20, if $\geq 20$ samples in day	NR 149.44(7)(d)1, (e)2-3	48
Limits ( $\pm 10\%$ ), failures, qualification and raw data	NR 149.44(7)(f-i)	48-49
<b>Limits of Detection and Quantitation</b>		
Determined annually	NR 149.48(2)(a-b), (d-g)	54-55
<b>Method Blank</b>		
1/preparation batch	NR 149.48(3)(a-b)	55
Detection, control limits and failures	NR 149.48(3)(c-d)	55
<b>Laboratory Control Sample, Source 2</b>		
1/preparation batch	NR 149.48(4)(a, d, e)	55
Calculations, control limits and failures	NR 149.48(4)(g-i)	56
<b>Matrix Spike and Matrix Spike Duplicate, Source 1 or 2</b>		
Only required if specified in method or in place of LCS	NR 149.48(5)(a)1 & 3	56
1/preparation batch and quality system matrix	NR 149.48(5)(b)2	56
Calculations, control limits and failures	NR 149.48(5)(c-d)	57
<b>Replicates</b>		
Only required if specified in method or in place of MSD	NR 149.48(6)(a-b)	57
Can use in place of MSD if results >LOQ	NR 149.48(6)(a)	57
1/ preparation batch and quality system matrix	NR 149.48(6)(c)	57
Calculations, control limits and failures	NR 149.48(6)(d-e)	57
<b>Quality Control Samples</b>		
Only if a source 2 spiking solution is not used for LCS, MS, MSDs	NR 149.48(8)	58
<b>Documentation and Labeling of Standards &amp; Reagents</b>		
Example: Ammonia standard source, concentration, purity, manufacturer, lot number, date of receipt and expiration	NR 149.45(1-2)	49
<b>Support Equipment</b>		
Thermometer, annual verification to NIST-traceable	NR 149.44 (3)(d)	43
Record refrigerator temperature (sample storage) and set to maintain proper temperature non-working (weekends, etc.) hours	NR 149.44(3)(e & f)	43-44
Analytical balance, monthly, in gm and mg-range	NR 149.44(3)(g)	44
Weights, NIST-traceable, 3-year external certification	NR 149.44(3)(g)1 & 2	44
<b>Sample Collection, Handling, and Storage</b>		
Sample collection records	NR 149.46(1)(a)	49
Container cleanliness	NR 149.46(1)(b)	50
Sample acceptance policy	NR 149.46(2)	50
Sample handling protocols & storage	NR 149.46(3) & (4)	50-52
<b>Data Reporting</b>		
Reported directly to WDNR	NR 149.47(1)(d)	52
Report elements, reported to client	NR 149.47(1)(e)	52-53

## SAMPLE AMMONIA BENCHSHEET

**Example 1:** Laboratory uses ISE meter in direct read mode, verifies slope with toggled meter function

### Meter Calibration

Standard	Volume, mL	[NH3], mg/L
STD 1	100	0.20
STD 2	100	2.00
STD 3	100	20.00

Analysis Date: 12/02/2004

Slope: -58.3

### Routine Analysis- Direct Read

Sample	Sample, mL	Dilution Factor	NH3, mg/L	QC %	Comment
Method Blank	100	1	0.01		
LCS, 1 mg/L	100	1	0.98	98%	
SAMPLE 1	100	1	5.60		
SAMPLE 2	100	1	0.12		
Closing CCV, 1 mg/L	100	1	1.02		

CCV is from same stock as calibration standards, LCS is from second source; laboratory not required to analyze QCS.

**Example 2:** Laboratory can only capture mV readings with meter, calculates results with computer.

### Meter Calibration- mV transformation

Analysis Date 12/02/2004

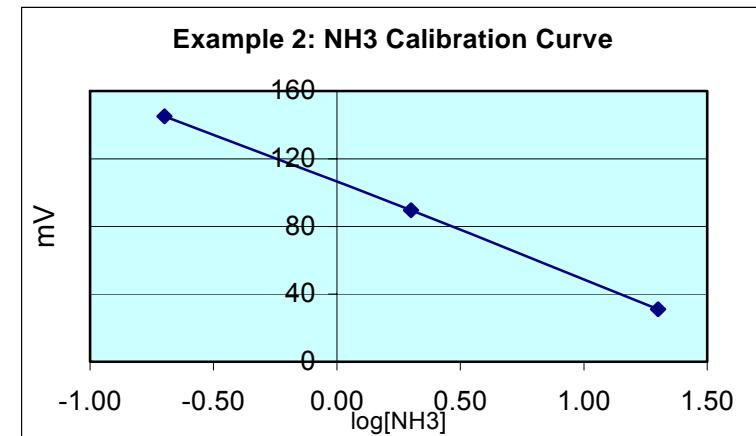
Standard	Volume, mL	[NH3], mg/L	log [NH3]	mV	Slope:	
STD 1	100	0.20	-0.70	145.1		-57.05
STD 2	100	2.00	0.30	89.5	Intercept	105.71
STD 3	100	20.00	1.30	31		

### Routine Analysis- mV transformation

Sample	Sample Volume, mL	Dilution Factor	mV	log <sup>-1</sup> [NH3]	Ammonia, mg/L	QC %	Comment
Method Blank	100	1	212	-1.86	0.01		
LCS, 1 mg/L		1	104	0.03	1.07	107%	
Sample 1	100	1	100	0.10	1.25		
Sample 2	100	1	226	-2.11	0.01		
Closing CCV, 1 mg/L	100	1	105	0.01	1.03	103%	

CCV is from same stock as calibration standards, LCS is from second source; laboratory not required to analyze QCS.

Method does not require spikes or replicates



## PROPOSED TOTAL PHOSPHORUS REQUIREMENTS

Activity, QC Element and Details	Cross Reference	Page
<b>Proficiency Testing</b>		
Renewal: 1 annually, analyzed between 9/1 and 8/15	NR 149.24(3)(a & b)	31-32
<b>Initial Demonstration of Capability</b>		
Exempted if combination of 4 LCS, MS, MSDs, PTs, QCS and Replicates analyzed in year prior to effective date	NR 149.36(3)(a)1	36
<b>Quality Manual</b>		
General provisions, format, content and revisions	NR 149.37	37-38
<b>Standard Operating Procedures and Analytical Methods Manual</b>		
SOPs can be copies of published methods followed exactly, with changes and additions specified or developed in-house	NR 149.40(1)	41
Analytical methods manual can be list of SOPs; part of QA manual	NR 149.40(2)	41-42
<b>Initial Calibration, Source 1</b>		
3 standards, minimum, at least annually	NR 149.44(6)(d)	45
R≥ 0.995 for linear	NR 149.44(6)(f)1 & (g)2	45-46
<b>Initial Calibration Verification, Source 2</b>		
Immediately after calibration, ±10% —OR—	NR 149.44(6)(i-j)	46-47
Exempted if Quality Control Samples analyzed	NR 149.44(6)(i)2	46
<b>Continuing Calibration Verification, Source 1</b>		
At beginning and end of batch if no calibration that day —OR—at beginning of batch only, if no calibration and use CCV from the next batch in place of ending CCV —AND— after every 20 samples if ≥20 samples analyzed in day	NR 149.44(7)(e)1-3	48
Limits: ±10%, failures, qualification and raw data	NR 149.44(7)(f)1, (g-i)	48-49
<b>Limits of Detection and Quantitation</b>		
Determined annually	NR 149.48(2)(a-b),(d-g)	54-55
<b>Method Blank</b>		
1/preparation batch	NR 149.48(3)(a-b)	55
Detection, control limits and failures	NR 149.48(3)(c-d)	55
<b>Laboratory Control Sample, Source 2</b>		
1/preparation batch	NR 149.48(4)(a)	55
Calculations, control limits and failures	NR 149.48(4)(g-i)	56
<b>Matrix Spike and Matrix Spike Duplicate, Source 1 or 2</b>		
Only required if specified in method or in place of LCS	NR 149.48(5)(a)1 & 3	56
1/preparation batch and quality system matrix	NR 149.48(5)(b)2	56
Calculations, control limits and failures	NR 149.48(5)(c-d)	57
<b>Replicates</b>		
Only required if specified in method or in place of MSD	NR 149.48(6)(a-b)	57
1/preparation batch and quality system matrix	NR 149.48(6)(c)	57
Calculations, control limits and failures	NR 149.48(6)(d-e)	57
<b>Quality Control Samples</b>		
Only if a source 2 spiking solution is not used for LCS, MS, MSDs	NR 149.48(8)	58
<b>Documentation and Labeling of Standards &amp; Reagents</b>		
Example: Hach PhosVer source, purity, concentration , manufacturer, lot number, date of receipt and expiration	NR149.45(2)	49
<b>Support Equipment</b>		
Thermometer, annual verification to NIST- traceable	NR 149.44 (3)(d & e)	43
Record refrigerator temperature (sample storage) and set to maintain proper temperature non-working (weekends, etc.) hours	NR 149.44(3)(f)	43
Analytical balance, monthly, in gm and mg-range	NR 149.44(3)(g)	44
Weights, 3 year verification	NR 149.44(3)(g)2	44
<b>Sample Collection, Handling, and Storage</b>		
Sample collection records	NR 149.46(1)(a)	49
Container cleanliness	NR149.46(1)(b)	50
Sample acceptance policy	NR 149.46(2)	50
Sample handling protocols & storage	NR 149.46(3) & (4)	50-52
<b>Data Reporting</b>		
Reported directly to WDNR	NR 149.47(1)(d)	52
Report elements, reported to client	NR 149.47(1)(e)	52-53

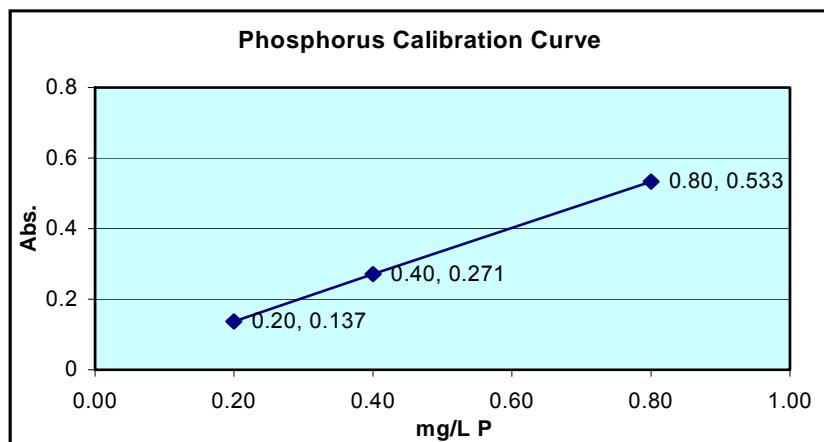
## SAMPLE TOTAL PHOSPHORUS BENCHSHEET

**Initial Calibration**

Created  
6/25/04      Expires  
6/25/05

Sample	Phosphorus, mg/L	Absorbance
STD 1	0.20	0.137
STD 2	0.40	0.271
STD 3	0.80	0.533

6/25/2004, which included ICV and LCS from second source.



This is one week's samples, using the calibration curve generated 6/25/04 for quantitation.

**Routine Analysis**

Analysis Date 7/2/04

Sample	Sample (mLs)	Final Vol, (mLs)	Dilution Factor	Absorbance	Phosphorus, mg/L	% Recovery
Method Blank	50	50	1	0.001	-0.01	
CCV, 0.5 mg/L	50	50	1	0.319	0.47	94.0%
LCS, 0.6 mg/L	50	50	1	0.419	0.63	105.0%
Sample 1	5	50	10	0.485	7.27	
Sample 2	50	50	1	0.111	0.16	
Sample 3	5	50	10	0.369	5.51	
Sample 4	50	50	1	0.222	0.33	
Sample 5	5	50	10	0.345	5.14	
Sample 6	50	50	1	0.199	0.29	
Closing CCV, 0.5	50	50	1	0.298	0.469	93.8%

The CCV is from the same source as calibration standards and is read twice, at the beginning and end; LCS is from second source.

Because laboratory uses second source for LCS, it is exempt from QCS.

The method does not require the analysis of spikes or replicates.